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USE OF METALLURGICAL VARIABLES AND SURFACE PROPERTIES TO CONTROL HYDROGEN EMBRITTLEMENT

H. W. Pickering

Department of Materials Science and Engineering
The Pennsylvania State University
University Park, Pennsylvania 16802

The Pennsylvania State University

University Park, Pennsylvania

THE PENNSYLVANIA STATE UNIVERSITY

College of Earth and Mineral Sciences

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This final report summarizes research carried out during the course of a program directed toward evaluating the factors affecting the absorption of hydrogen by iron and steels during exposure to aqueous solutions. The effects of deposits, barrier-type layers and certain electrolyte additions were investigated. An add-on project was directed toward evaluating the mechanism of protection of CulONi alloy in simulated seawater.

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H. W. Pickering

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INTRODUCTION

As an important approach to prevent hydrogen-induced cracking in metals, this project's goal was to evaluate the methods for reducing the absorption of hydrogen in metals and, in particular, in steels when exposed to aqueous solutions. At the time this project started, it was well known that certain additions to the electrolyte, known as poisons, greatly increase hdyrogen absorption and that certain metals, e.g., Ni, could be effectively used as barrier-type coatings on steel to reduce hydrogen uptake. Otherwise little was known about either electrolyte or alloy conditions which could affect the absorption step itself, in particular in the direction of reducing hydrogen absorption, or about the factors, diffusivity and solubility of hydrogen, important in the permeability of hydrogen in nickel and other candidate barrier-type layers at ambient temperatures.

A broad based program for studying this problem would include the effects of compositional changes at the surface produced from either the solution or alloy sides of the interface, as well as of local changes in the electrode potential. The latter had been recently shown to vary greatly within pits in iron during anodic polarization (1), so that this possibility during cathodic polarization and open circuit corrosion could not be overlooked. Such a program would also include the study of coatings. Metal coatings are used commercially for preventing hydrogen entry into steel, e.g., austenitic stainless steel and nickel. In this study metal and oxide coatings were investigated with emphasis on the former. The virtual absence of information on the solubilitiy and diffusivity of hydrogen at room temperature in metals which are candidate barrier layers is mainly due to inadequate sensitivity and/or resolution of experimental techniques, e.g., the hydrogen permeation technique

was inadequate at the time for metals like nickel whose permeability for hydrogen is several orders of magnitude lower than for iron. An improved technique was needed.

Research proceeded along two directions. These were:

(a) Examination of the factors which affect the ratio of hydrogen absorbed by the metal to that evolved as H_2 gas. These factors include electrolyte and metal compositions and electrode potential at the local site of the hydrogen evolution reaction. Different metal compositions at and specific deposits on, the surface were produced by variation of the alloy composition and by ion implantation and electroplating.

(b) A study of adhesion of continuous metal and certain oxide coatings, and of the permeability, including the development of a modified hydrogen permeation method for the measurement of the diffusivity and solubility of hydrogen in the coatings.

For both (a) and (b) the main experimental approach was to use a hydrogen permeation cell in which hydrogen discharges from aqueous solution and absorbs on one (entry) surface of a metal membrane, diffuses through the membrane and oxidizes under appropriate experimental conditions at the other (exit) surface.

RESULTS AND CONCLUSIONS

Hydrogen Absorption

A theoretical study was initiated to explore the possibility that certain deposits on the surface would decrease hydrogen absorption, thereby producing the opposite effect to the well known poisons which increase hydrogen absorption. The main prediction of the study was that deposits with higher

hydrogen exchange currents than the substrate surface would decrease absorption if the hydrogen evolution mechanism on the substrate surface involves a dependency of the hydrogen coverage on the overpotential (2-5). Such a dependency has been generally accepted to exist for hydrogen evolution on iron. Hydrogen absorption was determined for three metals meeting this exchange current criterion, Pt, Cu and Ni, and for two different application methods, electrodeposition (2,5) and ion implantation (3,4). The data were consistent with the concept, and hydrogen absorption and permeation were reduced in all cases.

Systematic polarization and permeation data on the effects of Cl^- ion and H_2S gas in the charging solution were initiated and are currently being completed. Chloride ion and H_2S , which have opposite effects on absorption, have similar effects on the overpotential for hydrogen evolution.

A theoretical study was initiated on the mass transport kinetics of a solute, e.g., H, diffusing through a bilayer membrane. Solutions available at the time were all based on analogous solutions in the heat transfer literature. The implicit assumption in these solutions is that the concentrations of the solute is the same on both sides of the interface. A solution was not available in the heat transfer literature for the general case in which the concentration differs on either side of the interface (often by a significant amount) since temperature, its counterpart in the heat transfer calculation, is the same on both sides of the interface. This mass transport problem has been solved and has direct application to hydrogen diffusion through bilayer membranes (6), as well as more generally in physical metallurgy.

One of the advantages of the above modeling solution is for using permeation data to obtain the diffusivity and solubility of hydrogen in metals in which their product is very low. The latter is a characteristic of all barrier-type layers. Preliminary data of this kind have been obtained for Sn and Cd (5) and measurements are currently underway for Ni and certain other barrier-type metals.

Oxide coatings were prepared on steel by sputtering and by selective oxidation of solute, e.g., Zr, Ti, Al, and Si. Adhesion was found to be inadequate for the sputtered oxides under typical laboratory, hydrogen-charging conditions and only marginally acceptible of those formed by selective oxidation. While adherent, the oxides were quite good as barriers to hydrogen absorption (7).

Local Conditions of Electrode Potential and Solution Composition

In order to see whether or not large changes in electrode potential occurred in cavities during cathodic polarization, both theoretical and experimental studies were initiated. The incentive was the earlier observation of large electrode potential variations in pits in iron during anodic polarization (1).

Model solutions for the concentration profiles of the ionic species and for the electrode potential have been obtained (8-10). These results give semi-quantitative information needed for understanding mechanisms and kinetics of electrode reactions in local regions, e.g., cracks.

Experimental measurement of the electrode potential, solution composition and hydrogen gas formation in slots in iron during hydrogen charging showed (8,9,11)

(i) hydrogen gas bubbles readily formed and remained in the slot.

(ii) the local electrode potential shifted in the noble direction with the magnitude of the shift increasing as the bubbles formed.

(iii) iron dissolution became a thermodynamic possibility as E became more noble than the iron reversible potential, and it was observed to occur at the base of the slot.

Similar results were shown for Ni and Cu, except that the limiting potential for Cu is less than its reversible potential and, consistent with this thermodynamic condition, no Cu dissolution was observed in the slot during hydrogen charging (8,9,12).

Considering the magnitude of the measured shifts of electrode potential and the theoretical limiting values for both anodic and cathodic polarization, it becomes clear that the electrode potential for gas-filled cavities remains virtually independent of the potenital at the surface even for large polarization in either direction (8-12).

Cu-Ni Alloy Corrosion in Simulated Seawater

Though not an original goal of the project, work was initiated on the mechanisms of protection of Cu10Ni alloy in 3.4 wt.% NaCl solution. The following aspects of the mechanism were determined from electrochemical and spectroscopy methods (13,14):

(i) the anodic metal dissolution reaction rate increases sharply with potential and is mainly determined by the diffusion of cations in the electrolyte within the porous corrosion product at potentials in the vicinity of the corrosion potential.

(ii) The cathodic reduction rate of oxygen is weakly dependent on electrode potential and is much suppressed by the poor catalytic nature of outer (porous) corrosion product layers.

(iii) The low corrosion rate in 3.4 wt.% NaCl and, therefore, probably also for seawater is due to (ii).

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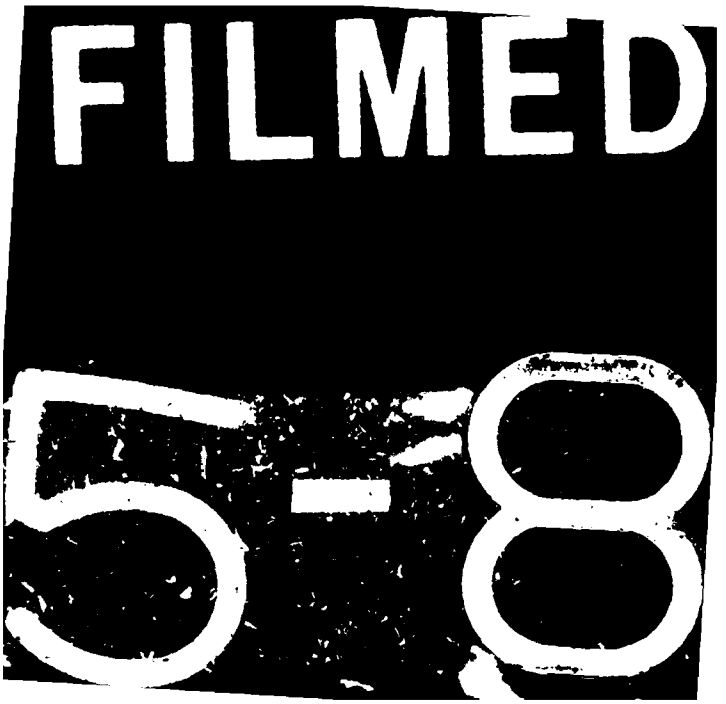
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